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**APPLICATION
FOR
UNITED STATES LETTERS PATENT**

Title: A Method, and Article of the Method, for Fabricating a Flexible,
Hollow Waveguide.

Inventors: Veena Gopal
James A. Harrington

3

4 Field of the Invention:

5 The present invention relates to waveguides and methods
6 of making same, and more particularly to a flexible, rugged
7 waveguide comprising a photonic, bandgap, hollow fiber,
8 transmitting in the infrared and visible regions.

10

BACKGROUND OF THE INVENTION

12

13 The flexible waveguide of this invention is based upon
14 technology disclosed in United States Patent Nos. 5,815,627;
15 issued: September 29, 1998; 5,567,471, issued: October 22,
16 1996; and 5,440,664; issued: August 8, 1995, which technology
17 and teachings are meant to be incorporated herein by way of
18 reference. The aforementioned waveguide inventions have a
19 common inventor, James Harrington, and a common assignee,
20 Rutgers University. In aforementioned United States Patent No.
21 5,815,627, it was taught that waveguides are capable of
22 guiding both IR and visible radiation. This is also a
23 distinguishing feature of this invention. This invention,

24 however, has fabricated a waveguide that can be tailored for
25 use over a wide wavelength range by the use of multiple,
26 uniform, film coatings deposited by liquid phase chemistry
27 methods. The coatings of the inventive waveguide have an
28 additive, are fabricated by an additive technique rather than
29 a subtractive method, i.e. the former Ag layer as taught in
30 the aforementioned patents, was originally depleted in part,
31 with the establishment of the AgI layer. The AgI layer is
32 totally absent in the present application. Even the Ag layer
33 is only present in one embodiment thereof, and is but one
34 selection of other metals used for the metallic underlay.

35

36 This invention utilizes dielectric layers of sulfide
37 materials, which are built in stacked geometries to fashion
38 unique waveguide structures.

39

40 The waveguides of the present invention can be fabricated
41 in one of two novel ways:

42

43 (a) The dielectric layers of cadmium and lead
44 sulfide can be applied directly to the smooth
45 bore surface of a silica glass tube; or

46

(b) The dielectric layers of the cadmium and lead sulfides can be applied to a metallic surface, for example, metals selected from a group consisting of Ag, Au, Cu, Pt, Ni, Pt, Mb, and Al, which has been smoothly coated upon the smooth inner bore of the silica glass tube.

53 A protective polymer coating is layered upon the outer
54 surface of the tube in both embodiments. The dielectric
55 layers of this invention comprise CdS and PbS, which form a
56 unique compatibility pair, in which deposition of each
57 material does not affect the underlying layer. These paired
58 sulfides can be stacked in multiple, thin film layers to
59 provide specific and unique characteristics. The sulfide
60 layers are deposited using similar liquid-phase chemistry
61 taught in the prior patents. The thickness of each layer can
62 be tailored for use over a wide range of wavelengths. The
63 measured losses for a single layer of CdS or a PbS film
64 deposited over an Ag layer was in agreement with the prior
65 Ag/AgI film studied at 10.6 μ m for applications using CO₂ IR
66 lasers.

69 The CdS and PbS materials have disparate refractive
70 indices with a ratio of about 2 to 1. This is often referred
71 to in this technology, as the index contrast ratio. The high
72 contrast of this layering makes possible a photonic bandgap
73 hollow fiber. The deposit of the cadmium sulfide layer
74 provides the transmission in the visible region.

75

76 Discussion of Related Art:

77

78 Prior-art hollow waveguides can provide poor beam quality
79 (poor transverse spatial coherence). FIG. 12B of Gregory &
80 Harrington, and FIGS. 5 and 6 of Croitoru et al.,
81 "Characterization of hollow fibers for the transmission of
82 infrared radiation", Appl. Opt. v. 29, 1805-1809 (20 Apr.,
83 1990) and Dror et al., "Hollow Tubes for Transmitting IR Laser
84 Energy for Surgery Applications", presented to ICALEO '89
85 (15-20 Jan., Los Angeles), are representative of the
86 characteristics of prior art hollow waveguides.

87 United States Patent Nos. 5,815,627; issued: September
88 29, 1998; 5,567,471, issued: October 22, 1996; and 5,440,664;
89 issued: August 8, 1995, depict Ag/AgI film waveguides.
90 Typically, waveguides such as are disclosed in Matsuura &
91 Miyagi, "Low-loss metallic hollow waveguides coated with

92 durable and nontoxic ZnS", Appl. Phys. Lett. v. 61, 1622-1623
93 (5 Oct., 1992) is superior. Waveguides with metal tube walls
94 that serve as the supporting structure for any coatings (such
95 as disclosed in U.S. Pat. No. 5,005,944, issued to Laakman et
96 al., and U.S. Pat. No. 4,913,505, issued to Levy '505) may be
97 capable of handling substantial power, but are semi flexible
98 at best. Those with plastic tube walls (such as disclosed in
99 U.S. Pat. No. 4,930,863, issued to Croitoru et al.) are
100 flexible, but have marginal power-handling capability at best
101 and high loss. Yet-earlier devices such as disclosed in U.S.
102 Pat. No. 3,436,141, issued to Comte, U.S. Pat. No. 3,583,786,
103 issued to Marcatili, and U.S. Pat. No. 3,963,828, issued to
104 Onoda et al., have not proven useful for the applications of
105 present interest at the wavelengths of present interest.

106

107 Hollow waveguide fibers having an index of refraction
108 less than one, have not yet attained both transmission
109 characteristics and flexibility required for many
110 applications. However, in other respects these waveguide
111 fibers are quite satisfactory at selected wavelengths. See
112 Gregory & Harrington, "Attenuation, modal, and polarization
113 properties of $n < 1$, hollow dielectric waveguides", Appl. Opt.
114 v. 32, 5302-5309 (20 Sept., 1993).

115
116 As aforementioned, the current invention is an
117 improvement over the waveguide devices of the prior aforesaid
118 Harrington patents.

119
120 The present invention has made loss measurements at key
121 laser wavelengths, e.g. as a CO₂ laser waveguide. The current
122 invention provides a flexible, hollow, waveguide, and method
123 for making same. The waveguide tube meets the need for a
124 flexible, visible and IR region, laser transmission medium
125 having a relatively low loss.

126
127
128
129

SUMMARY OF THE INVENTION

131
132 In accordance with the present invention, there is
133 featured a flexible, hollow waveguide for the transmission
134 of radiation in the infrared and visible regions, and a
135 method of making same. The waveguide comprises a hollow,
136 flexible, silica-glass tube having a transparent annular
137 body defining a bore with a smooth inner bore surface. In

138 a first embodiment, a reflective, thin film, metallic
139 layer, consisting of a metal selected from a group of
140 metals consisting of: Ag, Au, Cu, Ni, Pt, Mb, Zn, and Al,
141 is coated upon the smooth inner bore surface of the silica-
142 glass tube. A single, thin film cadmium sulfide dielectric
143 layer, or a pair of thin film dielectric, cadmium and lead
144 sulfide layers respectively, are then disposed upon said
145 reflective layer. Cadmium sulfide transmits radiation in
146 both the visible and infrared region. This pair of sulfide
147 layers has disparate refractive indices with a ratio of
148 approximately 2 : 1, which is vital to provide high
149 contrast, and to fabricate a photonic, bandgap, hollow
150 waveguide tube. In a second embodiment, no metallic layer
151 is used, and the pair of thin film cadmium and lead sulfide
152 layers is multiply stacked directly upon the smooth, inner
153 bore of the silica-glass tube.

154

155 The thin films are deposited using dynamic wet chemistry,
156 and the thickness is tailored to minimize the attenuation
157 of the waveguide over specific infrared wavelengths.

158

159 It is an object of the present invention to provide a
160 improved flexible, hollow waveguide.

161 It is another object of this invention to provide a
162 flexible, hollow waveguide comprising a photonic, bandgap
163 hollow fiber.
164
165 It is yet a further object of the current invention to
166 provide a flexible, hollow waveguide, which transmits
167 radiation in both the visible and infrared region.
168

BRIEF DESCRIPTION OF THE DRAWINGS

177 A complete understanding of the present invention may be
178 obtained by reference to the accompanying drawings, when
179 considered in conjunction with the subsequent detailed
180 description, in which:

181
182 FIGURE 1 depicts the growth kinetic graph of the thin
183 films deposited in the Ag coated 1,000- μ m bore waveguide;

184
185 FIGURE 2 shows the graph of the UV-VIS spectra of a
186 1,000- μ m bore Ag/CdS waveguide
187
188 FIGURE 3 illustrates the graph of the FTIR spectra of a
189 1,000- μ m bore Ag/Pbs waveguide;
190
191 FIGURE 4 depicts a graph of the cross-sectional FESEM
192 image of a 1,000- μ m bore Ag/CdS/PbS waveguide;
193
194 FIGURE 5 shows a graph of the FTIR spectra of a 1,000- μ m
195 bore of Ag/CdS and Ag/PbS, Ag/CdS/PbS and Ag/CdS/PbS/CdS
196 waveguide;
197
198 FIGURE 6 illustrates a partial schematic view of the
199 first embodiment of the waveguide of this invention;
200
201 FIGURE 6a depicts a partial schematic view of the second
202 embodiment of the waveguide of this invention;
203
204
205

205 DESCRIPTION OF THE PREFERRED EMBODIMENT

206
207 Generally speaking, a flexible, hollow, waveguide is
208 featured, that functions in the infrared and visible regions.
209 The waveguide comprises a hollow, flexible, silica-glass tube
210 having a smooth bore that is coated in one embodiment with a
211 reflective, metal substance on the inner bore surface. A pair
212 of sulfide materials respectively of cadmium and lead sulfide,
213 is then layered over the reflective substance. The sulfide
214 materials form a high contrasting refractive index of
215 approximately 2 : 1, thus creating a photonic, bandgap tube.
216 In a second embodiment, the metallic layer is not used, and
217 the pair of sulfide layers is singularly or multiply stacked
218 directly upon the bore.

219
220 There are two embodiments of the invention. The first
221 embodiment fabricates the waveguide by liquid phase deposition
222 of a metallic, reflective layer, usually Ag upon the smooth
223 inner bore surface of the silica-glass tube. Other metals of
224 choice such as Au, Cu, Ni, Pt, Zn, Mb, Al, etc., can also be
225 used. Then, a single or pair of sulfide-containing
226 dielectrics is coated over the metal. In the second
227 embodiment of the invention, no metallic or reflective layer

228 is deposited, and only pairs of sulfide-containing dielectric
229 films are coated upon the smooth inner bore.

230
231 Now referring to FIGURE 6, a first embodiment of the waveguide
232 1 of this invention is illustrated. The waveguide 1,
233 comprises a silica-glass tube 3, having a thin-wall of
234 approximately 50 to 200 microns wall thickness, and a smooth
235 inner bore. A reflective, metallic layer 4 is coated over the
236 smooth bore using liquid phase chemistry, as taught in the
237 aforementioned patents. The reflective layer may comprise Ag,
238 Au, Ni, Cu, Al, Pt, Zn, Mb, etc. A single layer of cadmium
239 sulfide, or paired sulfide composite of cadmium sulfide and
240 lead sulfide, respectively, is layered over the reflective
241 layer 4. The sulfide composite comprises respective layers 5
242 and 6, of cadmium and lead sulfides. The sulfide materials
243 form a high contrast, refractive index of approximately 2 : 1
244 creating a photonic, bandgap tube. The silica-glass tube 3 is
245 covered with an outer layer 2 of plastic for protection.

246
247 Referring to FIGURE 6a, a waveguide 10 is shown having a
248 flexible, hollow, silica-glass tube 11, upon which is stacked
249 at least one pair of cadmium and lead sulfide layers 12, and
250 14, respectively. A multiplicity of pairs of cadmium and lead

251 sulfide layers 12 and 14 can be deposited over the first pair,
252 as shown in phantom.

253

254 **Examples:**

255

256 1. A smooth, inner bore surface 3 of a flexible
257 hollow tube 2 of a waveguide 1, is coated with a
258 metallic, reflective layer 4 of silver. The
259 silver layer 4 was then coated with cadmium
260 sulfide to form layer 5, and then coated with
261 lead sulfide to form layer 6. Each layer was
262 coated using liquid phase chemistry, utilizing a
263 peristaltic pump. The flow rate of the
264 solutions through the hollow tube was chosen at
265 30 ml/min.

266 The cadmium sulfide coating was applied over
267 the Ag coating, using one of the two chemical
268 baths.

269 1. I) Cadmium Nitrate 0.1 M, II) thiourea 0.5 M -
270 this is considered a full concentration
271 solution. For most depositions, the
272 concentration was reduced to 1/2 to 1/6 the
273 full concentration.

274 2. I) Cadmium acetate 5mM, II) thiourea 100mM.

275
276 All the coating solutions were prepared in
277 distilled and deionized water. The solutions are
278 prepared using an ultrasonicator or a magnetic
279 stirrer.

280 The Cd ion containing solution is complexed
281 with ammonium hydroxide solution. Initial addition
282 of ammonia will form a white precipitate of Cd(OH)₂
283 and the solution becomes turbid. With further
284 addition of ammonia dissolves the white precipitate
285 and forms a soluble Cd-ammonia complex. The solution
286 pH is maintained between 10 and 13 using sodium
287 hydroxide solution and nitric acid as titrating
288 agents. The second solution of sulfide ion is
289 prepared by dissolving thiourea in water. The two
290 solutions were then flowed through the hollow tube
291 and coated the Ag. The complexed Cd ion solution
292 reacts with the sulfide ion containing solution to
293 deposit thin film of CdS. The hollow tube was dried
294 in flowing air.

295 2. For deposition of PbS thin films, lead nitrate was
296 used as the source of Pb ions and thiourea or a mix of

297 thiourea and thioacetamide was the source of sulfide
298 ions. All the solutions were prepared in distilled and
299 deionized water (DI). The solutions are prepared using
300 an ultrasonicator or a magnetic stirrer. The chemical
301 bath used for the deposition of lead sulfide is as
302 given below:

303 Solution 1: A) $\text{Pb}(\text{NO}_3)_2$: 4g / 500 ml; b) NaOH: 12
304 g/500ml and

305 Solution 2: thiourea: 6g/ 1000 ml or thiourea 4g and
306 thioacetamide 2 g to 1000ml of water

307 The NaOH solution is slowly added to the $\text{Pb}(\text{NO}_3)_2$
308 solution while continuing to stir. Initially the
309 solution turns turbid due to the precipitation of
310 $\text{Pb}(\text{OH})_2$ which dissolves to form a soluble Na-Pb-
311 hydroxide complex on further addition of NaOH. The
312 complexed lead ion reacts with a sulfide ion in
313 solution to form PbS. Deposition of PbS requires that
314 the solutions containing Pb and S ions are
315 supersaturated in order to precipitate out in solution
316 and nucleate heterogeneously on the substrate. The
317 bore surface of the waveguide was coated with the
318 solutions using the peristaltic pump as previously
319 shown. Surfaces were air dried.

320
321 3. Infrared and visible region investigation of Ag/CdS,
322 Ag/PbS, Ag/CdS/PbS and Ag/CdS/PbS/CdS waveguides, as
323 can be observed with reference to the FIGURES 1
324 through 3 and 5.
325
326 In the original work on omni directional waveguides authored by
327 Fink, et al.^[3] an all-dielectric structure of alternating low/high
328 index films was described. In this invention, use of a metallic
329 film in conjunction with a multilayer dielectric stack means that
330 the waveguide will need fewer dielectric layers to achieve the same
331 loss as an all dielectric omni directional structure. CdS and PbS
332 films are transparent in the 2 to 12 μm region. The refractive
333 indices of CdS and PbS are 2.25 and 4.27 at 1.55 μm and 2.25 and
334 4.0 at 10.6 μm , respectively. This gives an index contrast of
335 $4.27/2.25 = 1.9$ at 1.55 μm and 1.78 at 10.6 μm . These two
336 dielectric materials and other sulfides such as ZnS and ZnSe may be
337 deposited in thin film form using straightforward solution
338 chemistry methods^[4-6]. Furthermore, these two materials are
339 compatible, and the wet chemistry methods used to deposit both
340 films are similar^[7,8].
341

342 Dielectric-coated metallic hollow waveguides of this invention are
343 designed to minimize the attenuation of the waveguide over a
344 particular IR wavelength region by optimizing the thickness of each
345 dielectric layer. The well-established theory of Miyagi and
346 Kawakami^[9] was used to calculate the expected losses for multilayers
347 of CdS and PbS coatings. Using this theory, and the n and k values
348 for CdS and PbS, the losses for single and multilayer films at both
349 1.55 and 10.6 μm were calculated. The calculations at 10.6 μm show
350 that a 1,000- μm bore of a hollow glass waveguide with a 3-layer stack
351 of CdS/PbS/CdS films deposited over Ag will have a straight loss of
352 0.016 dB/m compared to a straight loss of 0.07 dB/m for a single
353 layer CdS film deposited over Ag. That is, the calculated
354 attenuation coefficients are approximately four times less for the
355 three layer design, compared to a single dielectric layer at 10.6 μm
356

357 As aforementioned, hollow glass waveguides are prepared in a
358 two step process in which an Ag film is first deposited on the
359 inner surface of the silica tubing and then a dielectric layer of
360 CdS or PbS is deposited on top of the metallic layer. For
361 multilayer dielectric structures, sequential deposition of
362 alternating low/high films leads to the structures, Ag/CdS,
363 Ag/CdS/PbS, and Ag/CdS/PbS/CdS. The Ag film is deposited on the
364 inner surface of the silica tubing using a liquid-phase reduction

reaction typical of that used in the prior patents to Harrington using Ag/AgI hollow glass waveguides^[10-12]. The thickness of the Ag film is chosen to be sufficiently thick to prevent any transmission through the film, but thin enough so that the surface roughness is as low as possible. Specifically, the thickness of the Ag film is chosen to be at least ten times the skin depth at 10.6 μm . The skin depth of an Ag film at 10.6 μm is 12 nm. In all the experiments CdS and PbS have been deposited on Ag films that vary in thickness from 150 to 200 nm. At this thickness the Ag films are quite smooth (< 12 nm rms roughness) and, therefore, they provide a good surface for the deposition of the dielectric layers. A very important feature of the CdS and PbS coatings is that they are additive and, thus, independent of the Ag film. This is in strong contrast to the waveguides made using AgI coatings^[11,13,14]. AgI is deposited in a subtractive process in which AgI is formed by the diffusion of iodine ions into the underlying silver film. Therefore, to produce a thick dielectric layer of AgI will require a thick starting layer of Ag. In the article by Rabii and Harrington^[15] have shown that the surface roughness of both the Ag and the AgI film increases with increasing thickness of the Ag layer.

386

387 **Additional Examples:**

388

389 As aforementioned, CdS and PbS thin films were deposited using
390 a wet chemistry deposition technique^[4]. Cadmium nitrate and
391 cadmium acetate are used as the source of Cd ions; lead nitrate
392 the source of Pb ions; and thiourea, ($\text{SC}(\text{NH}_2)_2$), the source of S
393 ions. It is important to accurately control pH of these
394 solutions, since the CdS and PbS precipitates are stable only in
395 the pH range 10 to 13. pH control and chelating is achieved
396 using an ammonia solution for CdS and sodium hydroxide for PbS.
397 Specifically, the pH for aqueous $\text{Cd}(\text{NO}_3)_2$ is maintained between 11
398 and 12. Owing to the fact that the reaction rate also changes
399 with the pH, it is necessary to calibrate the process for a given
400 pH range. The thickness of both CdS and PbS increases with
401 increasing concentration of Cd, Pb and S ions in solution and
402 with increasing deposition time. From a study of the growth
403 kinetics for both CdS and PbS thin films, it has been
404 established, that the optimal concentration and flow rate
405 conditions for uniform film deposition for tubing require lengths
406 greater than 1.5 m. The best coatings were made using > 0.01 M
407 solutions and pumping rates of 30 ml/min.

408

409 The multilayer dielectric structures of Ag/CdS/PbS and
410 Ag/CdS/PbS/CdS, were prepared in a manner similar to the single-

411 layer dielectric metallic waveguides. The different layers were
412 coated in a sequential manner with an intermediate drying step
413 after coating each layer. An Ag-only tube was coated with a
414 single layer of either CdS, or PbS, when the 2- and 3-layer
415 structures were being coated as an independent check on the
416 thickness for each layer.

417

418 Optical characterization of Ag/sulfide film Waveguides

419

420 The spectral characteristics of the Ag/CdS and Ag/PbS hollow
421 glass waveguides were evaluated using a Perkin Elmer UV-VIS
422 spectrometer, and Nicolet Protégé FTIR. A typical spectral
423 response shows interference peaks, which depend on the thickness
424 of the dielectric thin films. The thickness of a single layer
425 dielectric, "d", was calculated from the peak position of the
426 longest-wavelength interference band, $\lambda_p^{(m)}$, using the relation^[12],

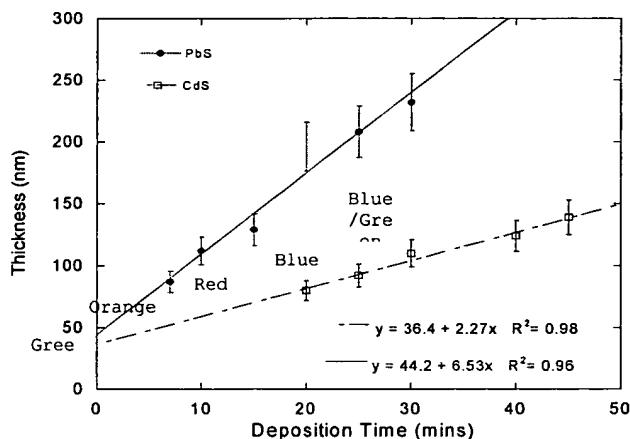
427

$$d = \frac{m \cdot \lambda_p^{(m)}}{4 \cdot \sqrt{n_1^2 - 1}},$$

428 where m is the order of the interference maxima ($m=1$ for the
429 longest-wavelength band); λ_p is the wavelength of the m^{th}
430 absorption peak; and n_1 is the refractive index of the dielectric
431 film. From Eq. (1) it is observed, that the peak position
432 shifts to longer wavelengths as the thickness of the film

433 increases. Spectral data has been used to determine the
434 thickness of films prepared using different growth kinetics. The
435 film thickness obtained from the optical measurements has then
436 been correlated with direct thickness measurements using a
437 field-emission scanning electron microscope (FESEM). In this
438 way one obtains the growth kinetic curves for CdS and PbS
439 deposited on Ag, shown in Fig. 1.

440



441

442 Fig. 1 The growth kinetic curves of the CdS and PbS thin films
443 deposited in a Ag coated, 1,000 μm bore Hollow Glass Waveguide.

444

445 The color indicated is the color that one would see looking
446 through the waveguides with an optical microscope.

447

448 All films were prepared using 0.01 M solutions and a flow rate
449 of 30 ml/min. The growth kinetics curves in Fig. 1 indicate that

450 Cds (open symbols) has a slower growth rate on Ag compared to PbS
451 (solid symbols) under similar conditions. The reason for the
452 slower growth rate for Cds compared to PbS, is that the Cds
453 complexes with the ammonia used in the deposition and this
454 decreases the rate of deposition. The data also shows that the
455 thickness of both dielectrics increases linearly with time, with
456 growth rates of ~ 2.3 and 6.9 nm/min for Cds and PbS,
457 respectively. The mechanism for linear growth is based on the
458 Stransi-Krastanov model of island-like growth^[16]. This mechanism
459 involves nucleation and growth in the linear growth region. This
460 information is important as we need to carefully control film
461 thickness for single and multilayer structures. The UV-VIS
462 spectra for the Ag/Cds is given in Fig. 2., and the FTIR spectra
463 for the Ag/PbS in Fig. 3. The spectral data clearly show that the
464 position of the interference peaks shifts to longer wavelengths as
465 the thickness of the film increases as predicted by Eq. (1). It is
466 also noted from the insert photos in Fig. 2, that the Hollow Glass
467 Waveguides show a color variation. This is due to selective
468 filtering of the input white light by the thin film coating
469 (interference effect). This color variation is seen with the Cds
470 coatings, but not the PbS films, because these films do not
471 transmit well at visible wavelengths.

472

The thickness of the thin films was obtained by direct measurements using a FESEM. A photomicrograph taken with the FESEM for a typical cross-section of the thin film combination Ag/CdS/PbS is shown in Fig. 4. The CdS film appears darker than the PbS film, because the in-lens detector produces a negative image of the secondary electron image. The thicknesses of the films shown in Fig. 4 are; Ag 154 nm, CdS 169 ± 16 nm, PbS 82 ± 6 nm. Degradation was not observed for the underlying film, when the new film is deposited over it. That is, CdS and PbS do not react with each other during the deposition of successive layers. The film thickness measured from the FESEM micrographs of both single and multilayer dielectric film are summarized in Table 2, below. These results agree very well with the optical thickness measurement.

487 Table 2 Thickness values for CdS and PbS thin films on Ag
 488 determined from FESEM images

HGWs	Thickness of		PbS
	Ag	CdS	
	nm	nm	nm
Ag/CdS	156	172 ± 16	—
Ag/PbS	158	—	96 ± 16
Ag/CdS/P	154	169	82 ± 6

489

490 A series of 1,000- μm -bore HGWs with 1, 2, and 3 dielectric layers
491 deposited over Ag were fabricated using wet chemistry methods [8,
492 11]. The spectral losses for these straight waveguides are shown
493 in Fig. 5. From Fig. 5, it may be seen that the addition of each
494 dielectric layer shifts the interference peaks to longer
495 wavelengths. This is a result of the increase in thickness with
496 each additional layer.

497

498 The thickness of each dielectric layer was determined from a wit
499 sample composed of each dielectric layer deposited separately on
500 These witness samples were deposited along with the multilayer wavegu
501 From the witness samples we determined the thickness of the indivi
502 layers using the position of the long wavelength interference peak
503 Eq. (1). The thicknesses obtained were; Ag 200 nm, CdS (adjacent to
504 156 nm, PbS 87 nm, and CdS (next to air) 97 nm. The thickness value
505 CdS and PbS layers measured optically agree very well with F
506 measurements and are within experimental errors as shown in Table 1.
507 Fig. 5 we see that this waveguide is best suited for operation beyo
508 μm .

509 Losses measurements were made at 1.55 μm using a diode laser rather
510 a CO₂ laser at 10.6 μm where the HGWs would ultimately be most use
511 The reason for using a 1.55 μm laser was that we were interested

512 developing a new waveguide for secure communication systems at 1.55
513 Clearly, solid-core silica fibers are a better choice for
514 applications at 1.55 μm . The output of the diode laser was vi
515 pigtailed single mode fiber terminated with a Selfoc lens. The spec
516 response of the HGWs chosen for loss measurements was similar to
517 shown in Fig. 5. In general, coating thicknesses were not optimized
518 lowest loss at 1.55 μm ; however, as may be seen from the cut-back
519 data given in Table 2, the losses were still quite low. The meas
520 losses in Table 2 may be compared to the theoretical losses calculate
521 both 1.55 and 10.6 μm using the n and k values of the dielectric f
522 and the theory of Miyagi and Kawakami [9]. The losses at 1.55 μm are
523 when PbS is used because k is rather large at this wavelength ($k = 0.$
524 At 10.6 μm k for PbS is 0.008 and the calculated loss for Ag/CdS/PbS
525 is over four times less than for Ag/CdS. The bending losses for
526 waveguides were not measured. It is well known that there is
527 additional loss on bending for non-omni directional waveguides, w
528 varies as $1/R$, where R is the radius of the bend [17]. Since
529 waveguides have no more than 3 dielectric layers it is expected tha
530 would not observe omni directional behavior rather there would be
531 added loss on bending.

532 Table 2 Loss values for 1,000- μm bore HGWs with 1, 2, and 3-layer
533 dielectric coatings.

534	4.	Multilayer	Measured l μm,	Theoretic 1.55 μ	Theoretic 10.6 μ	Conclusions summary
536	We	Ag/CdS	0.2 ±	2.7	7.0	have
		Ag/PbS	0.26 :	2.5	7.3	
		Ag/CdS/PbS	0.1 ±	8.6	3.3	
		Ag/CdS/PbS/Cc	0.06 :	6.7	1.6	

545 demonstrated that liquid-phase chemistry methods can be used to
 546 deposit good optical quality CdS and PbS thin films to form both
 547 single and multiple dielectric/metallic HGWs. The spectral response
 548 for waveguides with these films deposited over Ag show well defined
 549 interference bands indicating good film thickness uniformity over the
 550 entire length of the guide. Varying the deposition time controls the
 551 position of the interference peaks. Furthermore, we have found that
 552 CdS and PbS are compatible and the deposition of each material does
 553 not affect the underlying film. In this way the thickness of each
 554 layer in the multilayer stack can be tailored for use over a wide
 555 wavelength range. The final 3-layer stack showed that it is possible
 556 to make a multilayer coating but we have yet to reduce the
 557 attenuation to the level predicted by theory. Moreover, the measured
 558 losses for the single-layer CdS or PbS films at 1.55 μm are in
 559 general agreement with the well studied Ag/AgI HGWs at 10.6 μm [17].
 560 In reality, the greatest potential for these waveguides is likely to

561 be at 10.6 μm for applications involving CO₂ laser power delivery and
562 IR fiber sensors.

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564 References

- 565
- 566 1. E.D. Palik and G. Ghosh, Handbook of optical constants of
567 solids, (Academic, London, 1998).
- 568 2. Sopra database for n and k, <http://www.sopra-sa.com> (2003).
- 569 3. Y. Fink, J. N. Winn, S. Fan, C. Chen, J. Michel, J.
570 Joannopoulos, and E. Thomas, "A dielectric omnidirectional
571 reflector," *Science*, 282, 1679-1682 (1998).
- 572 4. R. S Mane and C. D Lokhande, "Chemical deposition method for
573 metal chalcogenide thin films," *Mat. Chem. Phys.* 65, 1-31
574 (2000).
- 575 5. P. K. Nair, M. T. S Nair, V. M Garcia, O. L Arenas, Y Pena, A
576 Castillo, I. T Ayala, O Gomezdaza, A Sanchez, J Compos, H Hu,
577 R Suarez, and M. E. Rincon, "Semiconductor thin films by
578 chemical bath deposition for solar energy related
579 applications," *Solar Energy Materials and Solar Cells* 52, 313-
580 344 (1998).
- 581 6. P. C Rieke and S.B. Bentjen, "Deposition of cadmium sulfide
582 films by decomposition of thiourea in basic solution," *Chem.*
583 *Mat.* 5, 43-53 (1993).

- 584 7. V. Gopal, and J. A. Harrington, "Dielectric coatings for
585 Ag/dielectric hollow glass waveguides," in Optical Fibers and
586 Sensors for Medical Applications II, Proc. SPIE 4616, 143-151
587 (2002).
- 588 8. V. Gopal, and J.A. Harrington, "Metal sulfide coatings for
589 hollow glass waveguides," in Optical Fibers and Sensors for
590 Medical Applications III, Proc. SPIE 4957, 97-103 (2003).
- 591 9. M. Miyagi and S. Kawakami, "Design theory of dielectric-coated
592 circular metallic waveguides for infrared transmission," J.
593 Light wave Technol. LT-2, 116-126 (1984).
- 594 10. M Mohebbi, R Fedosejevs, V. Gopal, and J. A. Harrington,
595 "Silver-coated hollow-glass waveguide for applications at 800
596 nm," Appl. Opt. 41, 7031-7035 (2002).
- 597 11. K. Matsuura, Y. Matsuura, and J. A. Harrington, "Evaluation of
598 gold, silver, and dielectric-coated hollow glass waveguides,"
599 Opt. Eng. 35, 3418-3421 (1996).
- 600 12. C. D. Rabii, D. J. Gibson, and J. A. Harrington, "Processing
601 and characterization of silver films used to fabricate hollow
602 glass waveguides," Appl. Opt. 38, 4486-4493 (1999).
- 603 13. T. Abel, J. Hirsch, and J. A. Harrington, "Hollow glass
604 waveguides for broadband infrared transmission," Opt. Lett.
605 19, 1034-1036 (1994).

- 606 14. R. Dahan, J. Dror, and N. Croitoru, "Characterization of
607 chemically formed silver iodide layers for hollow infrared
608 guides," Mater. Res. Bull. 27, 761-766 (1992).
609 15. C. D. Rabii and J. A. Harrington, "Measurement and control of
610 thin film uniformity in hollow glass waveguides," Opt. Eng.
611 38, 2009-2015 (1999).
612 16. M. Ohring, The materials science of thin films, (Academic
613 Press, Boston, 1992).
614 17. Y. Matsuura, T. Abel, and J.A. Harrington, "Optical properties
615 of small-bore hollow glass waveguides," Appl. Opt. 34, 6842-
616 6847 (1995).
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619 Since other modifications and changes varied to fit
620 particular operating requirements and environments will
621 be apparent to those skilled in the art, the invention is
622 not considered limited to the example chosen for purposes
623 of disclosure and covers all changes and modifications
624 which do not constitute departures from the true spirit
625 and scope of this invention.

626

627 Having thus described the invention, what is desired to
628 be protected by Letters Patent is presented in the
629 subsequently appended claims.

630

631 What is claimed is: